FATIGUE TESTING OF PLASMA-SPRAYED THERMAL BARRIER COATINGS

VOLUME 2

By

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FINAL REPORT

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1. Acknowledgments

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2 Plasma-Sprayed Bend Fatigue Testing

In addition to conducting the compression and tensile test program on the Pratt & Whitney EB-PVD coating (Volume 1 of this report), the cooperative agreement also required SwRI to assist Caterpillar Inc. in its developmental effort of a plasma-sprayed thermal barrier coating for diesel engines by providing test support. SwRI's task consisted of the fatigue screening of candidate thermal barrier coating materials and the generation of a data base for the selected candidate material.

2.1 Specimen Configurations

Bending fatigue specimens were supplied by Caterpillar Inc. in three different formats:

- 1. 0.75-inch X 4.75-inch substrates with coatings,
- 2. 0.38-inch X 3.00-inch substrates with coatings,
- 3. 0.375-inch X 2.15 X 0.085-inch average thickness coating material only (no substrates).

Respective thickness values of the specimens are reported in the tables detailing the test matrices.

2.2 Test Setup

Both the screening and the data base test matrices were conducted in four-point bending mode. The test fixtures were fabricated from 316 stainless steel and were configured as shown in Figure 1¹. The inner span of the fixture was fastened to the water-cooled, upper extension column, which in turn was attached to the stationary load cell of an 11-KIP MTS hydraulic test system. The outer span was supported by a stainless steel sphere, which was held on the centerline of the load train by a conical bore in the lower, water-cooled extension column. Application of the stainless steel sphere provided the necessary final alignment at the fixture-specimen contact points. The lower extension column was attached to the hydraulic actuator shaft of the testing system. A lower capacity, more sensitive load cell was inserted between the system's load cell and the upper extension column for tests requiring small amplitude loads; namely, for the 426-B test group.

Two load span configurations were used in the program, 40-mm (1.575-inch) and 80-mm (3.15-inch) total distance between outer load points, depending on the specimen configuration.

In the elevated temperature tests, heating of the specimens and test fixtures was accomplished with a lightweight, split, clam-shell furnace shown in the figure. The hot zone temperature of the furnace was controlled with a "K" type thermocouple positioned in the approximate center of the hot zone. Lightweight thermocouples, attached to the bend fixture components, were used to monitor the fixture temperatures to assure that thermal stability of the test system was achieved. It was assumed that the specimen was at the desired test temperature when all test fixture components reached thermal stability at that temperature.

2.3 Specimen Preparation

In the initial stage of the screening program, specimens were tested in the "as received" condition. After several consecutive substrate failures, it was determined that premature fatigue crack initiation was taking place at the sharp edges of the substrates or at surface defects on the face of the substrates.

¹ Figures and tables are in Section 3.

A surface preparation procedure was instituted which included rounding of the sharp substrate corners and polishing of the face of the substrates in steps to a final 1 micron finish. Use of this procedure extended the life of the substrates to the point where generation of coating failure became possible.

Although the polishing procedure greatly improved substrate performance, substrate failure remained a continuously bothersome problem. In order to further improve substrate performance, an attempt was made to shift the neutral plane of bending of the "composite" (substrate/coating) beam in the direction of the free substrate surface and thereby decrease stress amplitudes at the free surface of the substrate, where the fatigue failures occurred. This shift can be accomplished by reducing the substrate thickness. Removal of material from the substrate surface was accomplished using surface grinding procedures and post-grinding polishing. Disappointingly, reduction of the substrate thickness did not produce the anticipated improvements.

2.4 Test Matrix

A total of 97 specimens were tested in the program: 35 in the screening phase and 62 to generate a data base. Test specimen designations, along with respective specimen dimensions, fatigue load amplitudes, and failure data, are reported in Tables 1 through 8.

A compressive bending fatigue loading mode was specified by Caterpillar for both screening and data base tests. Six candidate materials were tested, as shown in the tables. For the data base generation, both tensile and compressive bending modes were utilized.

2.5 Test Procedure

The four-point bending fixture described above was utilized for both tensile and compressive bending modes. The difference between the two procedures was determined by the specimen orientation in the test fixtures. Tensile mode implies that the surface stresses in the coating on the specimen were of a tensile nature.

All screening tests were performed at an "R" ratio of 0.07, and the specimens were fatigued to failure without any additional measurements.

In the data base test matrix, a total of 17 specimens were designated for static bend fracture testing. The specimens designated for room temperature fracture tests were instrumented with strain gages to determine the static, elastic modulus values for the coating. Head displacement values were measured simultaneously with the strain gage outputs to obtain a compliance value for the load train. Since application of strain gages at 400° C was not feasible, measured head displacement values along with the previously determined compliance values were used to calculate the elastic modulus for the coatings. All static fracture tests were performed at a constant, 0.050-inch per minute, head displacement rate.

Fatigue testing was conducted at 0.07 and 0.60 "R" ratios and at the maximum frequency rate obtainable for the particular specimen group. It was determined early in the test program that variation in cyclic frequency had no detectable effect on the fatigue strength levels. Fatigue frequencies ranged between 1 and 15 Hz for the program.

In addition to substrate failures, ceramic coating failures due to high contact stresses were also a problem. Attempts to reduce the effects of the high contact stresses through the use of copper or aluminum "load spreader pads" were mostly unsuccessful, and the approach was abandoned.

Observations regarding individual specimen failure modes are presented in Tables 3 through 8.

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The second, major objective of this test program was to generate spalling-type failures observed in the actual engine tests. This objective was met with only partial success; only four failures of this type were obtained during the entire test program. It became clear that the substrate/coating specimen configuration tested in four-point bending mode was not the ideal experimental approach for generating this type of failure. After the desired data base information was obtained, further attempts to generate spalling-type coating failures were abandoned. It is suggested that alternative approaches, such as pre-compressed rotating beam fatigue testing, be investigated for producing such data.

2.6 Data Analysis

Test data analysis was performed entirely by Caterpillar personnel. Test results from individual tests were transmitted as they were generated, allowing Caterpillar personnel to select the most appropriate test parameters for the test to follow.

Partial test results generated during the program were presented by Randolph C. Brink of Caterpillar Inc. in the article, "Material Property Evaluation of Thick Thermal Barrier Coating Systems," published in <u>Transactions of the ASME</u>, <u>Journal of Engineering for Gas Turbines and Power</u>, Volume 111, Number 3, July 1989, pp. 570-577.

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3 Figures and Tables

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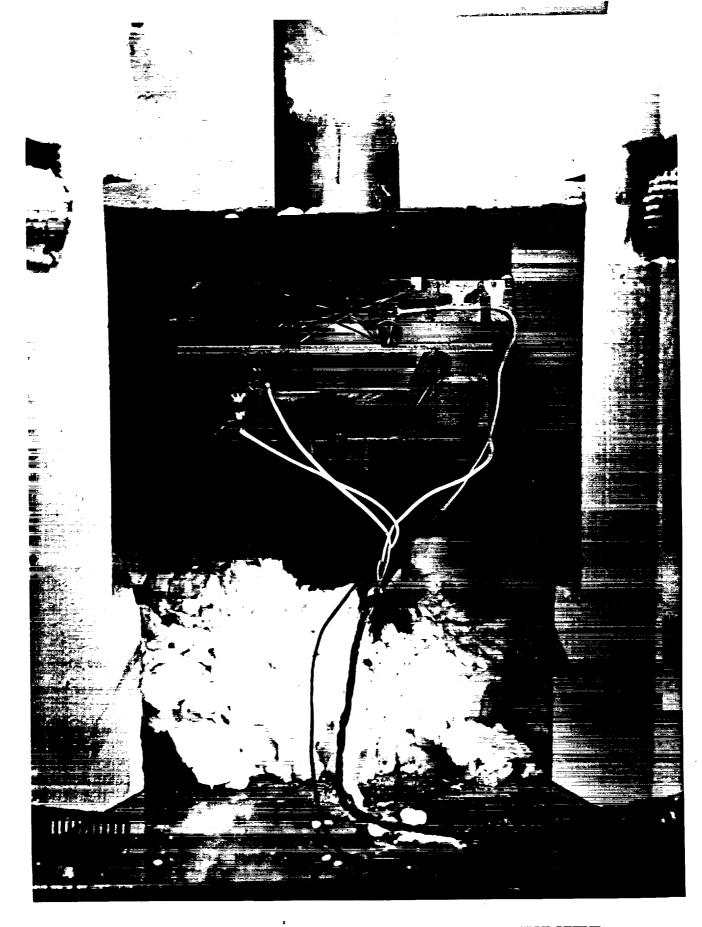


FIGURE 1. ELEVATED TEMPERATURE, FOUR-POINT BEND TEST SETUP

TABLE I SCREENING TEST SPECIMEN DIMENSIONS

		Thickness	
	,	(Inch)	
Specimen			
Number	Center	End #1	End #2
11001	Conto		
377-1	0.1700	0.1648	0.1663
377-2	0.1681	0.1653	0.1684
377-3	0.1699	0.1747	0.1641
377-4	0.1665	0.1645	0.1661
377-5	0.1709	0.1674	0.1682
377-6	0.1696	0.1738	0.1664
377-7	0.1690	0.1641	0.1675
377-8	0.1720	0.1719	0.1662
382-1	0.1531	0.1479	0.1514
382-2	0.1531	0.1479	0.1314
382-2	0.1522	0.1523	0.1441
382-3 382-4	0.1543	0.1323	0.1575
382-5	0.1543	0.1538	0.1543
382-6	0.1529	0.1532	0.1461
382-7	0.1493	0.1381	0.1487
382-8	0.1507	0.1481	0.1437
383-1	0.1518	0.1499	0.1449
383-2	0.1512	0.1556	0.1448
383-3	0.1510	0.1436	0.1516
383-4	0.1512	0.1486	0.1545
383-5	0.1506	0.1458	0.1541
383-6	0.1526	0.1472	0.1524
383-7	0.1493	0.1450	0.1478
383-8	0.1510	0.1492	0.1480

NOTE: Measurements were taken on the widthwise center of the specimens at spanwise center and at 0.250 inch from each end.

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TABLE II
SCREENING TEST SPECIMEN DIMENSIONS

		Thickness (Inch)	
Specimen Number	Center	End #1	End #2
1C-1	0.1683	0.1664	0.1685
1C-2	0.1680	0.1668	0.1673
1C-3	0.1666	0.1680	0.1683
1C-4	0.1679	0.1668	0.1681
1C-5	0.1681	0.1677	0.1685
2C-1	0.1380	0.1361	0.1380
2C-2	0.1373	0.1355	0.1373
2C-3	0.1353	0.1370	0.1379
2C-4	0.1372	0.1380	0.1385
2C-5	0.1354	0.1345	0.1357
2C-6	0.1358	0.1346	0.1353
3C-1	0.1193	0.1159	0.1198
3C-2	0.1183	0.1192	0.1197
3C-3	0.1200	0.1174	0.1196
3C-4	0.1186	0.1183	0.1191
3C-5	0.1196	0.1155	0.1177
3C-6	0.1200	0.1187	0.1188

NOTE: Measurements were taken on the widthwise center of the specimens at spanwise center and at 0.250 inch from each end.

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TABLE III

FOUR-POINT BEND SCREENING TEST RESULTS

Failure Modes and Locations	2 Places in center span Under right load pin; specimen end #1 Under inner load pins Under left load pin; specimen end #2 Under right load pin; specimen end #1 Under right load pin; specimen end #2 Under right load pin; specimen end #2	Under right load pin; specimen end #1 2 places in center span, under left load pin, 1 place in left load span; specimen end #2 2 places in center span; 1 place in right load span; specimen end #1 Substrate failure in center span Substrate failure under right load pin; specimen end #2 Substrate failure under left load pin; specimen end #2 Substrate failure, ander left load pin; specimen end #2 Not a substrate failure; 1 place in center span	1 place in left load span; specimen end #2 Under right load pin; specimen end #2 Under right load pin; specimen end #1 (twisted specimen) Under both inner load pins Under right load pin; specimen end #1 Under right load pin; specimen end #1
Cycles to Failure	N/A N/A 1/4 740 4,670 6,190	N/A N/A 2,740 38,050 211,080 156,050 141,840 105,300	N/A N/A 710 21,240 16,060 - 4,530
Minimum Fatigue Load (lb)	0 0 N/A 140 32 28 34	0 0 32 22 32 32 33	. 22 × 32 ° 0 ° 1 ° 23 ° 0 ° 1 ° 23 ° 23 ° 23 ° 1 ° 23 ° 23 ° 23 °
Maximum Fatigue Load (lb)	53 801 N/A 480 540 540 452	868 896 896 410 426 424 425	416 557/670 426 420 422 - 424
Test Rate	0.050/min 0.050/min 10 Hz 10 Hz 5 Hz 5 Hz 5 Hz	0.050/min 0.050/min 5 Hz 5 Hz 5 Hz 5 Hz 5 Hz 5 Hz	0.050/min 0.050/min 5 Hz 5 Hz 5 Hz - 5 Hz
Test Mode	Tens. Fracture Compr. Fracture Compr. Fatigue Compr. Fatigue Compr. Fatigue Compr. Fatigue Compr. Fatigue	Compr. Fracture Compr. Fracture Compr. Fatigue Compr. Fatigue Compr. Fatigue Compr. Fatigue Compr. Fatigue	Compr. Fracture Compr. Fracture Compr. Fatigue Compr. Fatigue Compr. Fatigue Not Tested Compr. Fatigue Not Tested
Test Temp (°C)	400 400 400 400 RT RT RT	400 400 8T 8T 8T	400 400 RT - RT
Specimen No.	377-1 377-2 377-3 377-4 377-5 377-6	382-1 382-2 382-3 382-4 382-5 382-6 382-7	383-1 383-2 383-3 383-4 383-5 383-7 383-6

TABLE IV

FOUR-POINT BEND SCREENING TEST RESULTS

Failure Modes and Locations	Outside of left load pin; specimen end #2 Outside of left load pin; specimen end #1	Multiple breaks in coating within center span Under left load pin; specimen end #2 2 places in center span, outside of right load pin; specimen end #1 1 place in center span, under right load pin; Specimen end #1 Under right load pin; specimen end #1 Under left load pin; specimen end #2	Under left load pin; specimen end #2 Under left load pin; specimen end #2 Inside left load pin; specimen end #2 Under both load pins Inside right load pin; specimen end #1 Inside left load pin; specimen end #1 Inside left load pin; specimen end #2
Cycles to Failure	N/A N/A 	40 910 2,890 210 N/A N/A	23,420 159,120 185,710 80,350 N/A N/A
Minimum Fatigue Load (lb)	00111	23 25 34 25 0	18 20 16 19 0
Maximum Fatigue Load (lb)	450 438 	343 348 345 347 402 430	267 269 267 270 452 454
Test Rate	0.050/min. 0.050/min. 	5 Hz 5 Hz 5 Hz 5 Hz 5 Hz 0.050/min.	5 Hz 5 Hz 5 Hz 5 Hz 6.050/min.
Test Mode	Compr. Fracture Compr. Fracture Not Tested Not Tested Not Tested	Compr. Fatigue Compr. Fatigue Compr. Fatigue Compr. Fatigue Compr. Fracture Compr. Fracture	Compr. Fatigue Compr. Fatigue Compr. Fatigue Compr. Fatigue Compr. Fracture Compr. Fracture
Test Temp (°C)	RT 	RY KY KY	RT R
Specimen No.	15.25 15.25 15.45	20:1 20:2 20:3 20:3 20:4 20:5 20:5	30-1 30-3 30-3 30-5 30-6

*There were signs of some erosion under the load pin.

TABLE V

DATA BASE FATIGUE SPECIMENS

	Comments on Failures	Under R-LP	Under L-LP	Multiple failure	Substrate failure	Substrate failure	Substrate failure	Runout	Substrate failure, ceramic @ L-LP	Ceramic @ R-LP, delaminated	Substrate failure, ceramic @ R-LP	Ceramic @ R-LP	Delamination outside R-LP	Specimen not tested	Failure in outside span & @L-LP;	radiused delam.	Ceramic @ R-LP & multiple in	midspan Ceramic @ L-LP
	Cycles to Failure	N/A	N/A	94,990	55,800	192,550	188,600	10,000,000	1,308,700	148,600	4,904,800	1,076	8,724,000	:	430,000+		2,894,900	5,340
	Minimum Fatigue Load (lb)	0	0	8	25	18	18	19	17	21	20	220	210	1	210	;	19	59
	Maximum Fatigue Load (lb)	583.00	286.00	356.00	354.00	250.00	250.00	250.00	250.00	300.00	275.00	398.00	350.00	ł	350.00		300:00	420.00
	Test Rate	0.050/min	0.050/min	SHz	SHz	SHz	2Hz	5Hz	5Hz	15Hz	15Hz	15Hz	15Hz	1	15Hz		15Hz	1-5Hz
	Test Temp (°C)	RT	RT										RT	1	RT	ļ	RT	RT
	Test Mode	Compr. Fracture	Compr. Fracture	Compr. Fatigue	Compr. Fatigue	Compr. Fatigue	Compr. Fatigue	Compr. Fatigue	Compr. Fatigue	Compr. Fatigue	Compr. Fatigue	Compr. Fatigue	Compr. Fatigue	NA	Compr. Fatigue		Compr. Fatigue	Compr. Fatigue
	End #2	0.1824	0.1835	0.1850	0.1773	0.1726	0.1813	0.1755	0.1776	0.1763	0.1769	0.1663	0.1783	Y V	0.1692	0001	0.1823	0.1802
Thickness (Inch)	End #1	0.1777	0.1833	0.1837	0.1754	0.1677	0.1766	0.1724	0.1743	0.1751	0.1755	0.1660	0.1741	ΑΝ	0.1662	2	0.1799	0.1787
	Center	0.1694	0.1811	0.1745	0.1688	0.1594	0.1631	0.1651	0.1675	0.1673	0.1695	0.1551	0.1652	A A	0.1570	2020	0.1693	0.1700
	Specimen Number	426-Ti-42	426-Ti-43	426-Ti-44	426-Ti-45	426-Ti-47	426-Ti-48	426-Ti-49	426-Ti-54	426-Ti-55	426-Ti-56	426-Ti-57	426-Ti-58	426-Ti-59	426-Ti-60	:	10-11-074	426-Ti-62

NOTE: Specimen substrates, starting with 426 Ti-49, were polished!

R-LP = right load pin

L-LP = left load pin

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TABLE V (CONCLUDED)

DATA BASE FATIGUE SPECIMENS

		Comments on Failures		1	Ceramic damage during polishing	Ceramic @ R-LP	Ceramic @ L-LP	Ceramic @ L-LP	Ceramic @ R-LP & delamination	Ceramic @ R-LP & in mid-span	Ceramic @ R-LP & in mid-span;	(O.L. @ rem)	Ceramic @ L-LP &	mid-span/aluminum pads	Ceramic @ R-LP/aluminum pads	Ceramic @ LP/copper pads (37.5 mm	<u>2</u>	Ceramic @ R-LP & center/copper	pads (37.5 mm R)	Specimen not tested	Specimen not tested	Specimen not tested	Ceramic @ R-LP/copper pads (150	mm R)/wide contacts
	Cycles	to Failure	N/A	N/A	1	1,400	1,640	2,378	83,170	1,235,000	1,504,700		823	;	N/A	N/A		480		1	1	ı	N/A	
	Min. Fatigue	Load (B)	0	0	ł	8	20	21	18	19	17		53	Ó	-	0		30		1	ŀ	ł	0	
	Max. Fatigue	Load (B)	400.0	378.0	ŀ	295.0	300.0	298.0	250.0	210.0	225.0		425.0	į	8./4	662.1		432.0		ı	ı	ı	700.8	
		Test Rate	0.050/min	0.050/min	1	15Hz	15Hz	5Hz	15Hz	15Hz	15Hz		1SHz		0.050/mm	0.050/min		15Hz		ı	ı	ı	0.050/min	
	Test	Temp (°C)	RT	RT	1	RT	RT	RT	RT	RT	RT		RT	į	K	RT		RT		RT	RT	RT	RT	
		Test Mode	Compr. Fracture	Compr. Fracture	Not Tested	Compr. Fatigue	Compr. Fatigue	Compr. Fatigue	Compr. Fatigue	Compr. Fatigue	Compr. Fatigue		Compr. Fatigue	ţ	Compr. Fracture	Compr. Fracture		Compr. Fatigue		NA	NA	NA	Compr. Fracture	
		End #2	0.1531	0.1468	Ν	0.1493	0.1510	0.1461	0.1510	0.1453	0.1500		0.1508		0.1412	0.1542		0.1487		Y Y	Ν	Ϋ́	0.1402	
Thickness (Inch)		End #1	0.1451	0.1383	Ϋ́	0.1471	0.1501	0.1430	0.1491	0.1400	0.1485		0.1482	007	0.1438	0.1523	,	0.1466		Y V	NA	NA	0.1397	
		Center	0.1470	0.1454	٧X	0.1465	0.1485	0.1445	0.1473	0.1359	0.1460		0.1459	0	0.1385	0.1521	1	0.1425		N A	ΝΑ	A A	0.1435	
		Specimen Number	426-17-21	426-17-22	426-17-23	426-17-25	426-17-26	426-17-27	426-17-28	426-17-29	426-17-30		426-17-31		426-17-32	426-17-34	!	426-17-35		426-17-36	426-17-37	426-17-38	426-17-39	

Specimen substrates, starting with 426 Ti-49, were polished! R-LP = right load pin L-LP = left load pin NOTE:

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TABLE VI

DATA BASE FATIGUE SPECIMENS

									Ţ,							der	ıder	1	<u> </u>	Ider		iple	gce.
	Comments on Failures			Faihre @ L-LP	Faihre @ R-LP Faihre @ R-LP	Failure @ approximate center	Faihtre @ R-LP	Faihre @ L-LP	30 min. soak @ 400F; test @ RT;	midspan failure		Failure @ L-LP)	Runout - no failure	Aged: 40 hrs @ 770F;	Aged: 40 hrs @ 770F; failure under	load point Aged: 40 hrs @ 770F; failure under	load point	oad point	Aged: 40 hrs @ 770F; failure under	load point; Machine did not shut off after	specimen failure; apparent multiple	loading points on specimen surface.
	ω υ	-	 								 4 S			_				ر م					'으
	Cycles to Failure	N N	X X	1,220	3.420	35,820	N/A	∀ N	N/A	Nodete	6 672 200	1.276	2,153	10,000,000	N/A	135,050	9,420,000	,	•	188,590	No data		
	Minimum Fatigue Load (lb)	0	0	25.0	0.49	0.56	0	0	0	2	950	1.05	1.09	0.99	0	1.06	1.14	13	1	6:0	0.84		
	Maximum Fatigue Load (lb)	10.30	11.16	00.6	9.9	8.00	19.30	19.70	17.00	15.50	13.50	15.00	15.60	14.20	19.20	15.14	16.25	00.71	20.01	12.50	12.00		
	Test Rate	0.050/min.	0.050/min.	10Hz	10Hz	10Hz	0.050/min.	0.050/min.	0.050/min.	10H2	10H2	10Hz	10Hz	10Hz	0.050/min.	2Hz	2 & 10 Hz	111,		2 to 10Hz	2 to 10Hz		
	Test Temp (°C)	RT	RT	R	Z	RT	400	6	RT	400	8 6	9	400	90	400	90	400	Ţ		RT	RT		•
	Test Mode	Tens. Fracture	Tens. Fracture	Tens. Fatigue	Tens, Fatigue	Tens. Fatigue	Tens, Fracture	Compr. Fracture	Tens. Fracture	Tens Fations	Tens. Fatione	Tens, Fatigue	Tens. Fatigue	Tens. Fatigue	Tens. Fracture	Tens. Fatigue	Tens. Fatigue	Tene Hatime	angm t ang t	Tens. Fatigue	Tens. Fatigue)	
	End #2	0.0848	2980.0	0.0893	0.0890	0.0894	0.0871	0.0885	0.0878	0.8860	0.0887	9980.0	0.0897	9680.0	0.0888	0.0870	0.0891	0.0886	20000	0.0843	0.0852		
Thickness (Inch)	End #1	0.0780	0.0865	0.0878	0.0888	0.0885	0.0862	0.0861	0.0856	0.0885	0.0882	0.0861	0.0896	0.0893	0.0877	0.0865	0.0881	0.0884	2000	0.0819	0.0844		
	Center	0.0816	9980.0	0.0882	0.0848	0.0889	0.0862	0.0879	0.0841	0.0885	0.0870	0.0848	0.0897	0.0882	0.0887	0.0852	0.0885	0.0884		0.0825	0.0835		
	Specimen Number	426-B-1	426-B-2	426-B-3	420-B-4 426-B-5	426-B-6	426-B-7	426-B-8	426-B-9	426-R-10	426-B-11	426-B-12	426-B-13	426-B-14	426-B-15	426-B-16	426-B-17	47K B 18	27.02	426-B-19	426-B-20		

NOTE: R-LP = right load pin L-LP = left load pin

TABLE VII

DATA BASE FATIGUE SPECIMENS

	Comments on Failures	"Fish-Scale" type failure approx. 1/8 in. from R-LP in midspan	Copper pads, friction & wear type failure at pads and substrate under load points	Failure at midspan/spallation type	Failure at midspan/spallation type	"Peel" type failure in midspan	Specimen not tested	Specimen not tested
	Cycles to Failure	N/A	42,439	493	2,030	N/A	l	ł
	Minimum Fatigue Load (1b)	0	45.5	42.0	39.0	0	ı	l
	Maximum Fatigue Load (1b)	939.6	650.0	614.0	550.0	888.0	1	1
	Test Rate	0.050/min.	S Hz	5 Hz	5 Hz	0.050/min.	ı	l
	Test Temp (°C)	RT	RT	RT	RT	RT	ł	l
	Test Mode	Compr. Fracture	Compr. Fatigue	Compr. Fatigue	Compr. Fatigue	Compr. Fracture	I	I
	End #2	0.1390	0.1401	0.1407	0.1340	0.1279	1	ţ
Thickness (Inch)	End #1	0.1388	0.1362	0.1352	0.1336	0.1273		1
	Center	0.1375	0.1286	0.1250	0.1334	0.1269	!	-
	Specimen Number	360-17-5	360-17-6	360-17-7	360-17-8	3C-38	3C-39	3C-40

NOTE: R-LP = right load pin

TABLE VIII

DATA BASE FATIGUE TEST RESULTS

		Thickness (Inch)								
					Test		Maximum	Minimum	مراتبك	
Specimen				Test	Temp	Test	Load	Load	10	
Number	Center	End #1	End #2	Mode	ွ	Rate	(lb)	(lb)	Failure	Comments on Failures
4										
25	0.1233	l	1	Compr. Fracture	RT	0.050/min.	760	0	NA	I
92	0.1247		I	Compr. Fracture	RT	0.050/min.	734	0	NA	ı
27	0.1240	ì	l	Compr. Fatigue	RT	1-5 Hz	20%	35.6	17,190	Blisters prior to failure; "good" failure
88	0.1247	ı	i	Compr. Fatigue	RT	1-5 Hz	280	40.6	35,000	Blisters @ 20,000; "good" failure
. 32	0.1244	1	ŀ	Compr. Fatigue	RT	5 Hz	522	36.5	33,190	Substrate failure/multiple initiation
33	0.1225	0.1240	0.1234	Compr. Fatigue	RT	SHZ	419	29.3	98,380	Substrate failure
ቖ	0.1234	0.1248	0.1248	Compr. Fatigue	RT	SHZ	403	28.2	120,240	Substrate failure
36	0.0762	0.0777	0.0780	Compr. Fatigue	RT	2 Hz	130	9.1	164,640	Ceramic failure at load point
38	0.0785	0.0788	0.0789	Compr. Fatigue	RT	2 Hz	139	6.7	158,060	Substrate failure

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4 Conclusions and Recommendations

The testing performed was not totally satisfactory. The four-point bend test set-up which is so common in ceramics testing has been found unacceptable as a means for defining the fatigue strength of the material for high cycle conditions. The failure mode of surface layer spallation, observed in the engine testing program, was demonstrated to be a real failure mode in compression tests, but was difficult to achieve in the four-point bend testing.

Stress conditions in four-point bend testing are quite nonuniform. The contact stresses are substantial at the surface and for the layers of material and interfaces below the contact point. Curvature effects in bending as well as tolerance effects between the specimen and the loading bars can cause additional local stress conditions which can induce premature static and fatigue failures.

Long life fatigue testing is particularly challenging for specimens with nonuniform loading and internal stress conditions. Failure modes include specimen modes as well as material modes. The test program found that the surface roughness and, possible, residual stress conditions on the metallic substrate were extremely sensitive parameters at the desired test conditions. Polishing the substrate had only limited success in removing premature, specimen configuration fatigue modes.

Stress gradients, in four-point bend testing, result in material response that is only strictly accurate for the bend test itself. Uniform stresses in a well-defined gage section are highly desirable conditions for strength and fatigue testing. This is particularly true for ceramic materials where defect sizes play a strong role in both static and fatigue strength models. The four-point bend test results can be expected to be dependent on the thickness of the ceramic and the size of the specimen. The test matrix did not permit this evaluation to be confirmed.

Unfortunately, the reason that the four-point bend specimen is widely used for ceramics testing is that the test arrangement is relatively inexpensive. Additionally, other test specimen configurations for ceramics have not been developed that fully address the technical limitations of the four-point bend test.

Some limited thought was given to other, more suitable test arrangements for ceramic coatings. Monolithic ceramic test specimens of a reasonable size are virtually impossible to fabricate without the use of a metallic substrate. However, coating of a substrate and then removing the substrate through chemical etching is a possible means of test specimen fabrication. Pure compressive testing of a uniform tube of ceramic poses the difficulty of achieving good load introduction at the specimen ends in order to avoid local fracture, as well as the influence of friction constraint at the platten-specimen interface.

Tension testing of ceramics is even more difficult due to stress concentrations in the regions of specimen grips or the transitions to the gage section, or both. The use of the "Brazil" specimen for tension testing has been shown successful in earlier ceramic coating testing at SwRI. The specimen has the shape and proportions of a "Tums" tablet and is loaded in compression. The specimen internal stress distribution is a nearly uniform tensile stress across the plane between the load contact points. Tensile static and fatigue strength results have been successfully achieved for this specimen.

Rotating beam, ceramic coated tubular specimens were considered for high-cycle fatigue testing. The rotating beam test also has some substantial drawbacks. One of the principal test condition requirements for the current program was for high, steady stress together with superimposed cyclic stresses. The rotating beam test arrangement would need to include a steady, prestress in the coated tube; no such standard rotating-beam test machine is available. Design and fabrication of such a test

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machine is possible, but is judged to be relatively expensive, particularly when considering the high temperature requirements. Additionally, the load transition from the substrate into the ceramic coating in the test section is likely to produce its own, unique fatigue failures.

In conclusion, therefore, the testing did achieve partial success in spite of the inherent problems of the four-point bend specimen configuration. The use of simpler, uniform-stress test specimens is recommended for material evaluation. Tubular compression specimens and the "Brazil" specimen for tension testing are recommended as alternatives to the bending specimen.

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